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## CaO sorbent stabilisation for CO<sub>2</sub> capture applications

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### Abstract

Large differences have been observed in CO<sub>2</sub> sorption behaviour of various commercial calcium oxide materials during multi cycle sorption tests. ICP analyses of these materials have shown a significant variance in magnesium impurities within these materials. Because it is well known that magnesium can stabilize CaO sorbents, optimization of CaO for CO<sub>2</sub> sorption has been performed by examining the effects of magnesium loading with respect to the pre-cursor, Mg-loadings and temperature treatments. Experiments have been performed in a fixed bed reactor at 650°C in the presence of steam. The experimental results show that it is possible to improve the cyclic stability significantly. Loading with a small amount of magnesium (~1wt%), followed by a high temperature treatment at 1000°C results in a performance which is very similar to the commercial material with high magnesium impurities. The CaO show a stable cyclic capacity of 19% from the 150<sup>th</sup> cycle until the 250<sup>th</sup> cycle. Finally, Sorption Enhanced-Steam Methane Reforming experiments are performed with the stabilized material, resulting in an increased CH<sub>4</sub> conversion to almost 100%.

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**Keywords:** Pre-combustion decarbonisation; CaO; CO<sub>2</sub> sorbent; Cyclic capacity; Cyclic stability

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### 1. Introduction

Calcium oxide is a promising candidate for high temperature capture of CO<sub>2</sub>. The material combines good sorption and regeneration kinetics with a high theoretical CO<sub>2</sub> capacity of 17.8 mmol CO<sub>2</sub> per g CaO. However, the biggest challenge is to overcome the high degradation rate of the material. In a pre-combustion decarbonisation scheme, the CO<sub>2</sub> is captured while the fuel is being reformed resulting in a high H<sub>2</sub> yield due to a shift in the reforming equilibrium. A typical system consists of two connected fluidised bed reactors. In the first reactor, fuel is reformed and CO<sub>2</sub> is captured by carbonation of CaO to CaCO<sub>3</sub> at around 650°C at elevated pressure (25 bar). The carbonation of CaO proceeds in two stages; a fast stage occurring in the surface layers, and a slower stage in which CO<sub>2</sub> has to diffuse through the dense carbonate layer to convert the bulk of the material [1]. Practical applications therefore cannot make use of the full capacity related to the bulk CaCO<sub>3</sub> formation.

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In the second reactor  $\text{CaCO}_3$  is regenerated to  $\text{CaO}$  at  $800^\circ\text{C}$  using steam yielding a concentrated  $\text{CO}_2$ /steam stream from which the water can easily be knocked-out. Regeneration can also be performed with in-situ oxy-combustion or  $\text{CO}_2$ , but the latter requires a much higher regeneration temperature. During the high temperature regeneration step sintering of the material occurs which leads to a reduction in the porosity and available surface area of the material. One possible solution to overcome the loss in effective capacity would then be to stabilise the  $\text{CaO}$  pore structure in some manner. Numerous approaches have been proposed in literature [2,3,4,5].

Abanades et al. [6,7] showed by pilot scale experiments and an economic assessment that  $\text{CaO}$  can also be used in post-combustion systems. These systems can have a lower efficiency penalty and lower additional costs than commercially available amine-based adsorption systems. As such, the results presented here have value to both pre- and post-combustion decarbonisation schemes.

The current work describes the research performed at ECN in the field of  $\text{CaO}$  stabilization for  $\text{CO}_2$  sorption applications. A method will be presented by which pure  $\text{CaO}$  with a high deactivation rate can be stabilised. An automated flow-through reactor system with off gas analysis is used to determine the breakthrough curves and hence the capacities of the materials. Furthermore, results of Sorption Enhanced Reforming (SER) experiments with the stabilised  $\text{CaO}$  are presented.

## 2. Experimental

Cyclic  $\text{CO}_2$  sorption-desorption experiments were performed on a fully automated flow-through laboratory scale test-rig as described previously by Reijers et al. [8]. At the outlet, online gas analysis is performed by both a Hartman and Braun Uras 14  $\text{CO}_2$  analyzer and an HP Micro GC. The powder is pelletized and crushed stepwise in an agate mortar and a sieve fraction of 0.212–0.425 mm is collected. 1 gram of sieve fraction is placed in a quartz reactor with an internal diameter of 10 mm resulting in bed heights between 15 and 20 mm.

Carbonation and regeneration are performed at  $650^\circ\text{C}$  and  $800^\circ\text{C}$  respectively at a pressure of 1 bara. During the carbonation step a gas flow of  $30\text{ ml}\cdot\text{min}^{-1}$  containing 0.29 bara  $\text{H}_2\text{O}$ , 0.15 bara  $\text{CO}_2$  and balance  $\text{N}_2$  is fed to the reactor during 60 minutes resulting in almost complete breakthrough. The flow-through principle of the test-rig gives the opportunity to generate  $\text{CO}_2$  breakthrough curves of the sorbent. Regeneration is started by simultaneously switching off the  $\text{CO}_2$  flow, increasing the steam and  $\text{N}_2$  flows and ramping to  $800^\circ\text{C}$ . These settings result in short regeneration times of approximately 15 minutes.

Initial  $\text{CO}_2$  capacity experiments were performed on four commercially available  $\text{CaO}$  materials; one from Aldrich (ReagentPlus, 99.9% purity, further denoted  $\text{CaO-a}$ ), two from Alfa Aesar which will further be denoted as  $\text{CaO-mp}$  (purity 99.95% Metals basis) and  $\text{CaO-hp}$  (Puratronic, 99.998% Metals basis) and one Reagent Grade from a for confidentiality reasons non disclosed manufacturer, denoted as  $\text{CaO-b}$ . No further pre-treatments were carried out on these materials, other than starting the experiment under desorption conditions.

Based on these initial results and further physical and chemical characterization of the samples described in paragraph 3,  $\text{CaO-mp}$  was used in the investigation of temperature and magnesium loading effects on sorption behaviour. Thermal treatments in air were performed by ramping with  $2^\circ\text{C}\cdot\text{min}^{-1}$  to a set temperature. Subsequently, the sample was kept at that temperature for 4 or 6 hours followed by a non-assisted cooling down to room temperature. Loading with Mg was performed using dry impregnation. Finally, the slightly wet sample was dried overnight at  $80^\circ\text{C}$  in air followed by a thermal treatment. Table 1 shows the wide range of samples prepared with variation in Mg-precursor, Mg-loading, and calcination before and after impregnation.

Sorption Enhanced–Steam Methane Reforming experiments were performed on the same test-rig. One (1) gram of aged  $\text{CaO-b}$  sorbent from previous experiments (250 cycles) is used in combination with 1 gram of a pre-commercial reforming catalyst. The experiment was carried out using a feed gas composition of 7.1%  $\text{CH}_4$ , 29%  $\text{H}_2\text{O}$  and balance  $\text{N}_2$  at a total flow of  $25\text{ ml}\cdot\text{min}^{-1}$  and a temperature of  $550^\circ\text{C}$ .

In order to get more insight in the relation between material properties and cyclic stability, physical and chemical characterisations were performed on  $\text{CaO-a}$  and  $\text{CaO-b}$ . Examination of the sample morphologies was performed with a JEOL JSM-6330F scanning electron microscope. Pore size distribution (PSD) and BET specific surface area data were obtained by  $\text{N}_2$  physisorption on a Coulter Omnisorp 360 after a thermal treatment at  $350^\circ\text{C}$  for 20 hours at a pressure  $< 10^{-5}$  bar. The phase compositions of the samples were analyzed by means of X-ray diffraction (XRD) analysis with a Bruker Advance D8 apparatus in Bragg Brentano mode with a  $2\theta$  range of  $10$ – $100$  degrees, and a

step size of 0.05 degrees  $2\theta$ . The chemical composition of both samples was examined by Inductive Coupled Plasma (ICP) analysis on a Varian Vista AX apparatus.

Table 1, Sample preparation overview of Mg doped CaO sorbents.

Sample	Calcination 1 [°C/h]	Mg amount [wt%]	Precursor	Calcination 2 [°C/h]
CaO-mp00				
CaO-mp02		0.5	MgCl <sub>2</sub>	600/4
CaO-mp03		0.5	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	600/4
CaO-mp04		0.5	Mg(CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O	600/4
CaO-mp05		0.5	MgSO <sub>4</sub> ·7H <sub>2</sub> O	600/4
CaO-mp06		0.0*		600/4
CaO-mp07	1000/6	0.25	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	600/4
CaO-mp08	1000/6	0.75	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	600/4
CaO-mp09	1000/6	1	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	600/4
CaO-mp10	1000/6	1.25	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	600/4
CaO-mp11	1000/6	5	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	600/4
CaO-mp12	1000/6			
CaO-mp13		1.0	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	1000/4
CaO-mp17	1000/6	1.0	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	1000/6
CaO-mp19	1100/6			
CaO-mp20		1.0	Mg(NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	1100/4
CaO-mp26		0.0*		1000/4

\* Impregnated with demineralised water only

### 3. Results and Discussion

Cyclic CO<sub>2</sub> sorption experiments of the four non-treated materials show some remarkable differences in behaviour as is shown in Figure 1. CaO-mp clearly shows the highest initial capacity of 8.3 mmol·g<sup>-1</sup> but this is overshadowed by the very fast deactivation as function of cycle number. By cycle 35, the capacity has decreased to only 2.2 mmol·g<sup>-1</sup>, corresponding with 26% of the initial capacity and 12% CaO utilisation, close to the 7-8% residual utilisation of many CaO sorbents after prolonged cycling [9].

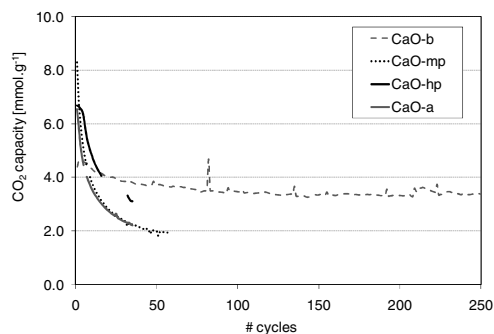


Figure 1, Cyclic CO<sub>2</sub> capacity of 4 commercial CaO materials.

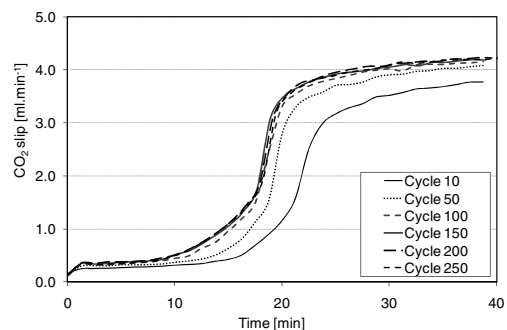


Figure 2, CO<sub>2</sub> breakthrough curves of CaO-b.

Although the initial capacity of CaO-a is significantly lower, the behaviour at increasing cycle number is similar to that of CaO-mp. CaO-hp starts with a comparable capacity as CaO-a but deactivates slightly slower. The most

striking result is that of CaO-b. The initial capacity is rather low but increases somewhat during the first few cycles which was also shown by Manovic and Anthony [3] and was attributed to the high temperature treatment. This small increase is subsequently followed by a rather slow decrease in capacity. After about 150 cycles of carbonation/regeneration this sorbent shows a stable performance at a capacity of about  $3.4 \text{ mmol} \cdot \text{g}^{-1}$ . The breakthrough curves of this experiment are displayed in Figure 2 from which it becomes obvious that stable operation is reached after about 150 cycles. This capacity represents 18.9% utilisation of the stoichiometric capacity, which is above the minimum of 15% suggested for high efficiency operation in SE-SMR [10].

The physical analysis of CaO-a and CaO-b revealed some striking differences (Table 2). In general, the results of CaO-a seemed more promising with a high BET surface area of  $22 \text{ m}^2 \cdot \text{g}^{-1}$  compared to only  $9 \text{ m}^2 \cdot \text{g}^{-1}$  for CaO-b. The higher surface area of CaO-a is a result of significantly higher porosity and smaller crystallites, which is also confirmed from SEM analyses (see Figure 3). However, these values are only reflected in the sorption properties of the first cycle(s). According to Alvarez and Abanades [1] carbonation takes place at a significant rate until a product layer thickness of 50 nm is reached. After this initial process diffusion limitations of  $\text{CO}_2$  through the dense carbonate layer decrease the reaction rate significantly. This indicates that the higher sorption capacity of CaO-a during the first few cycles may be attributable to the relatively high surface area. However, the difference in stability cannot be explained merely by the initial surface area, as CaO-a clearly deactivated much faster than CaO-b.

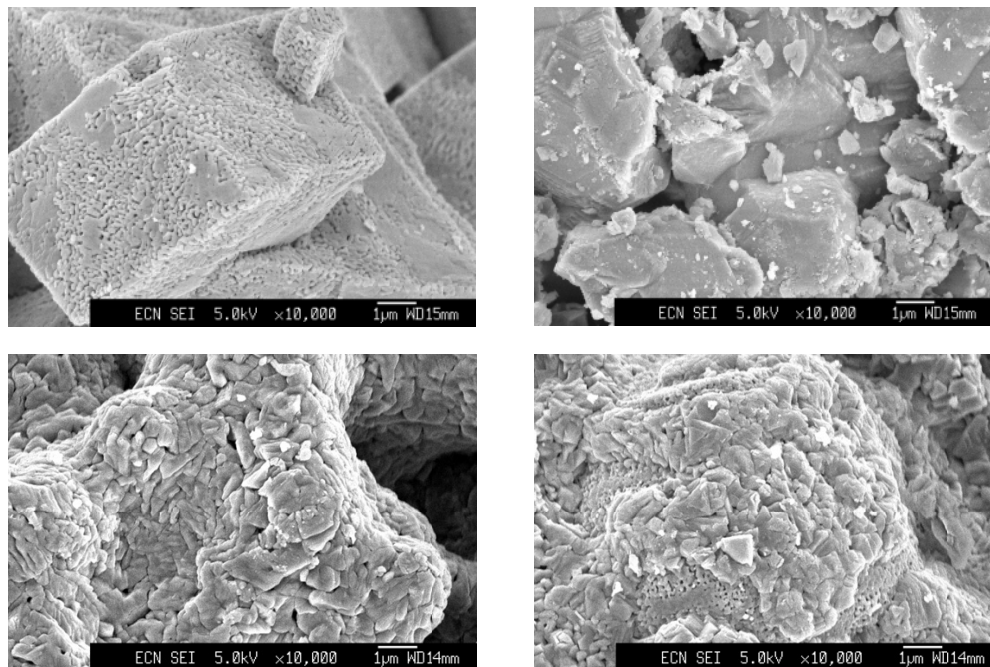


Figure 3, SEM images of CaO-a (left) and CaO-b right as fresh materials (top) and after 35 cycles of carbonation and regeneration (bottom).

PSD measurements of samples after 35 cycles show that the total volume of pores between 8–10 nm of CaO-a increases while the contribution of pores in the range of 20–40 nm decreases. During the carbonation process, the smallest pores are vulnerable to pore blocking by the increased volume of the carbonate layer [11]. Apparently, although the mesopores could be responsible for the high initial capacity, they were not stable under the conditions used here, resulting in a fast decrease of the capacity. This is in great contrast with CaO-b of which the pores smaller than 20 nm disappear while the pores  $>20 \text{ nm}$  stay intact, although the total pore volume decreases dramatically. This last observation would imply a low capacity but probably the nitrogen physisorption method is not representative for the  $\text{CO}_2$  chemisorption at  $650^\circ\text{C}$  in the presence of steam. Reduction of pore volume related to

the smaller pores and simultaneous increase of pore volume related to larger pores is described by Sun et al. [12,13] who used mercury intrusion which gives the opportunity of measuring larger pores. They indeed found an increase of the number of pores above 220 nm for Strassburg limestone after cyclic capture experiments without the presence of steam.

Table 2, Chemical and physical analysis results of CaO-a and CaO-b (numbers in brackets are after 35 cycles).

	CaO-a	CaO-b
BET S.A. ( $\text{m}^2\cdot\text{g}^{-1}$ )	20 (7.5)	8.7 (3)
Porosity (%)	15.4 (8.3)	6.1 (1.6)
PSD, main pores (nm)	20-40 (8-10)	10-20 (>20)
XRD pattern	bulk phase CaO, some $\text{Ca}(\text{OH})_2$	bulk phase CaO, some $\text{Ca}(\text{OH})_2$ and $\text{CaCO}_3$
Crystallites (XRD, Sherrer $k=0.9$ , nm)	35-45	135-150
ICP main impurities		
Al ( $\text{mg}\cdot\text{kg}^{-1}$ )	5	836
Fe ( $\text{mg}\cdot\text{kg}^{-1}$ )	2	499
Mg ( $\text{mg}\cdot\text{kg}^{-1}$ )	55	3296
Na ( $\text{mg}\cdot\text{kg}^{-1}$ )	25	260
S ( $\text{mg}\cdot\text{kg}^{-1}$ )	24	131
Si ( $\text{mg}\cdot\text{kg}^{-1}$ )	17	4364
Sr ( $\text{mg}\cdot\text{kg}^{-1}$ )	146	301

XRD analysis of both samples before the cyclic tests shows a bulk phase of cubic CaO where the crystallinity of CaO-b is slightly higher. No remarkable components are found besides the bulk phase and some hexagonal  $\text{Ca}(\text{OH})_2$  for CaO-b and both  $\text{Ca}(\text{OH})_2$  and rhombohedral  $\text{CaCO}_3$  for CaO-a. ICP chemical analysis confirms that CaO-a has a higher purity than CaO-b. The most conspicuous results are those of Mg and Si impurities. Where CaO-a only contains  $55 \text{ mg}\cdot\text{kg}^{-1}$  Mg and  $17 \text{ mg}\cdot\text{kg}^{-1}$  Si these values are 3296 and 4364  $\text{mg}\cdot\text{kg}^{-1}$  respectively for CaO-b. Albrecht et al. [2] investigated the effects of Magnesium loading on CaO looping stability, but in their work the lower limit of magnesium loading was 5wt% and the experiments were performed in the absence of steam. In this light, an experimental program was set up to examine the effects of lower amounts of magnesium loading on these sorbents. Additionally, the effect of temperature on Mg-doped sorbents was also examined.

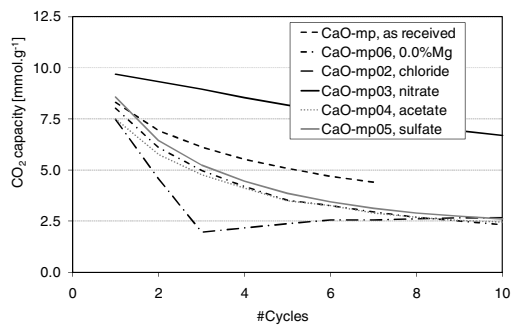


Figure 4, Effect of precursor on the cyclic stability after loading with 0.5wt% Mg.

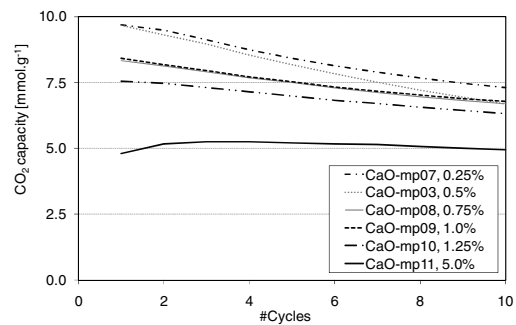


Figure 5, Effect of Mg concentration within the sorbent, based on  $\text{Mg}(\text{NO}_3)_2$ .

The effect of the Mg precursor is examined by loading with 0.5 wt% Mg on CaO-mp followed by calcination in air at  $600^\circ\text{C}$  for 4 hours. As shown in Figure 4, the deactivation of acetate and sulphate based samples had the same trend as CaO-mp06 which was a reference sample prepared with impregnation of demineralised water only.

The initial capacities of these aforementioned samples was slightly lower than the as received CaO-mp. CaO-mp02 based on  $\text{MgCl}_2$  behaved completely differently. Initially there is a very fast deactivation followed by a small re-activation and stabilization at around  $2.6 \text{ mmol}\cdot\text{g}^{-1}$ . CaO-mp03, doped with  $\text{Mg}(\text{NO}_3)_2$  shows the most promising behaviour, although the number of cycles is limited. The deactivation rate is still rather high but this sorbent starts with a high capacity of  $9.7 \text{ mmol}\cdot\text{g}^{-1}$  of which  $6.7 \text{ mmol}\cdot\text{g}^{-1}$  is left after 10 carbonation/regeneration cycles.

A search for the optimum Mg-loading was initiated with a series of experiments using various amounts of  $\text{Mg}(\text{NO}_3)_2$  on pre-calcined CaO-mp; this resulted in Mg-loadings of 0.25–5 wt%. As shown in Figure 5, Mg-loadings  $<0.75 \text{ wt}\%$  result in a high initial capacity of almost  $10 \text{ mmol}\cdot\text{g}^{-1}$  but is followed by a gradual loss of capacity to about  $7 \text{ mmol}\cdot\text{g}^{-1}$  at cycle 10. Increasing the Mg-loading to 0.75 and 1.0 wt% results in a slightly lower initial capacity while the capacity at cycle 10 is not affected. Further increasing to 1.25 wt% Mg does not affect the stability but decreases the overall capacity. A Mg-loading of 5% gives a relatively low initial capacity but little change in capacity during the first 10 cycles. Initial capacities and stability of this material were close to that of CaO-b.

The effect of a high temperature treatment, as suggested by Manovic and Anthony [3] on pure CaO, was examined by pre-calcining CaO-mp at both  $1000$  and  $1100^\circ\text{C}$ . Figure 6 shows that the stability is indeed slightly improved by this treatment. From literature [5,13] it is known that a steam or water treatment can enhance the pore structure and thereby the sorbent performance. CaO-mp26 is prepared by impregnation with demineralised water only in order to examine the hydration effect in combination with a high temperature sintering step. As shown in Figure 6, the small gain in stability by high temperature sintering is counteracted by the hydration, indicating that the experimental results obtained are probably due to the magnesium impregnation and post-calcination procedures only.

Subsequently, some  $\text{Mg}(\text{NO}_3)_2$  based samples were prepared with 1.0% loading and different pre- and post calcination treatments. Calcination at  $1000^\circ\text{C}$ , followed by impregnation and calcination at  $600$  or  $1000^\circ\text{C}$  results in materials with a relatively high capacity and noticeable deactivation during the first 10 cycles (Figure 7). In contrast, the samples prepared with Mg impregnation and post calcination only, behave differently resulting in the plot of capacity versus cycle number looking very similar to that of CaO-b.

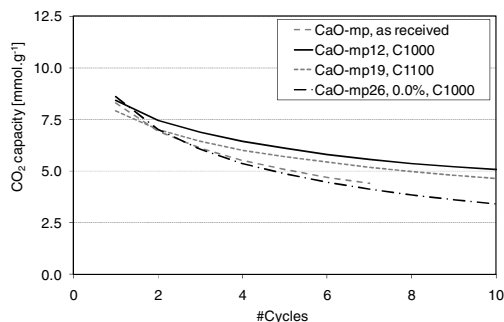


Figure 6, High temperature calcination effect on sorbent stability.

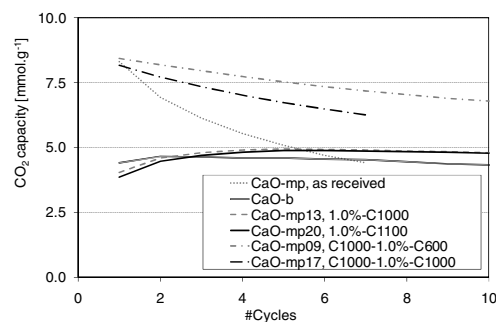
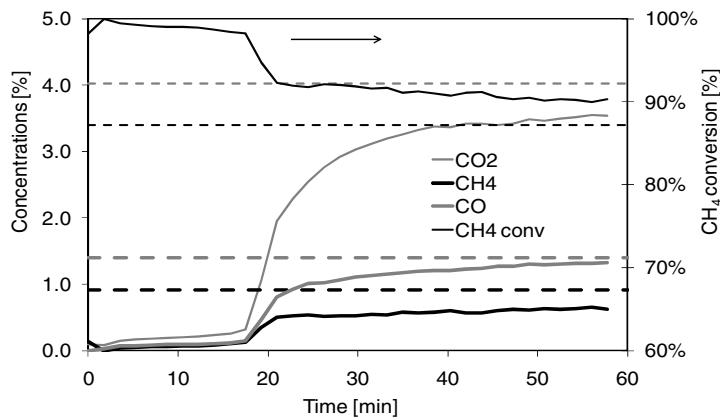


Figure 7, Mg loading and temperature effects on ten adsorption/regeneration cycles.

These results show that it may be possible to stabilise CaO with only a small effort. Results of the adapted materials are very close to those of as received CaO-a, meaning that stabilised materials are already available. Moreover this offers the opportunity of a relatively cheap modification of only 25\$ worth of added  $\text{Mg}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$  per ton of natural calcites. With the price of  $\text{CaCO}_3$  of only about 5\$/ton, the addition of 1% Mg has quite some effect. However, even with this modification, the sorbent costs would be around 30 \$/ton which is still 4 times cheaper than  $\text{Fe}_2\text{O}_3$ , the second cheapest material for chemical looping, per ton of captured  $\text{CO}_2$  [14]. Although these results might also be significant in other CaO- $\text{CaCO}_3$  looping cycles like post combustion, our goal final goal was to test its performance in a SMR experiment.

The result of the 10<sup>th</sup> SE-SMR cycle, as described in the experimental section, is shown in Figure 8, where the outlet concentrations for CO<sub>2</sub>, CH<sub>4</sub> and CO are plotted as well as the CH<sub>4</sub> conversion and the thermodynamic equilibrium values. It can clearly be seen that during the first 18 minutes the reaction was shifted beyond its thermodynamic equilibrium. The CH<sub>4</sub> conversion starts close to 100% which is about 13 percent points higher than the non-enhanced equilibrium. In the subsequent minutes the conversion decreases slowly to about 98% due to an increased saturation state of the sorbent. Subsequently, it drops to 92% followed by another gradual decrease towards the equilibrium value. This behaviour can completely be understood in terms of the known thermodynamics and kinetics of the interaction of CO<sub>2</sub> with CaO. The partial pressure of CO<sub>2</sub> slipping from the reactor should be below 2.4 mbar in order to obtain an enhanced methane conversion to 95%. CaO sorbents fulfil this requirement by a small margin at 550°C only. As soon as the fast carbonation stage has ended the CO<sub>2</sub> breaks through the sorbent bed and simultaneously the CH<sub>4</sub> and CO concentrations increase. Although experimental settings are different these results look similar to those described by Harrison [15].



**Figure 8.** Results of the 10<sup>th</sup> SE-SMR reforming cycle of CaO-b in combination with a pre-commercial reforming catalyst at 550°C.

#### 4. Conclusions

It is shown that a low purity commercial CaO sample behaves completely different with respect to cyclic CO<sub>2</sub> sorption stability in the presence of steam compared to a number of commercial pure CaO samples. ICP analysis indicated a large difference in magnesium content which is, based on general literature, a possible explanation for the improved stability. Stability of the commercial sample was reproduced with only a simple impregnation of Mg on CaO carriers. Initial experiments have been performed to investigate different Mg precursors of which Mg(NO<sub>3</sub>)<sub>2</sub> is the most promising with an increased initial stability and improved cyclic stability, although only 10 cycles were used. The other precursors do not show a significant improvement of sorbent properties, although no attempts was made to investigate this any further.

High temperature treatment at 1000 and 1100°C of pure CaO sorbents results in a slight improvement of stability but this increase is counteracted by a subsequent impregnation with demineralized water. Pre-calcination at 1000°C followed by loading with 1.0wt% Mg and post-calcination at 600 or 1000°C has little effect on sorbent properties compared to doping with 1.0wt% followed by calcination at 600°C. In contrast, loading with 1.0% Mg and calcination at 1000 or 1100°C completely changes the stability behaviour resulting in a great similarity to that of CaO-b.

SE-SMR is performed with a combined catalyst-sorbent bed with CaO-b and a pre-commercial reforming catalyst. Thermodynamic calculations of the steam methane reforming reaction show that CO<sub>2</sub> partial pressure down to below 2.4 mbar are required in order to obtain a CH<sub>4</sub> conversion above 95%. Although the carbonation reaction only just fulfils this requirement, sorption enhanced conversion is clearly observed. The thermodynamic equilibrium

CH<sub>4</sub> conversion under the given settings is 87% whereas during the SE period the conversion is shifted to values between 100 and 98%, depending on the saturation state of the sorbent.

## 5. Acknowledgements

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